

Separation Columns

(Distillation, Absorption and Extraction)

11.1. INTRODUCTION

This chapter covers the design of separating columns. Though the emphasis is on distillation processes, the basic construction features, and many of the design methods, also apply to other multistage processes; such as stripping, absorption and extraction.

Distillation is probably the most widely used separation process in the chemical and allied industries; its applications ranging from the rectification of alcohol, which has been practised since antiquity, to the fractionation of crude oil.

Only a brief review of the fundamental principles that underlie the design procedures will be given; a fuller discussion can be found in Volume 2, and in other text books; King (1980), Hengstebeck (1976), Kister (1992).

A good understanding of methods used for correlating vapour-liquid equilibrium data is essential to the understanding of distillation and other equilibrium-staged processes; this subject was covered in Chapter 8.

In recent years, most of the work done to develop reliable design methods for distillation equipment has been carried out by a commercial organisation, Fractionation Research Inc., an organisation set up with the resources to carry out experimental work on full-size columns. Since their work is of a proprietary nature, it is not published in the open literature and it has not been possible to refer to their methods in this book. Fractionation Research's design manuals will, however, be available to design engineers whose companies are subscribing members of the organisation.

Distillation column design

The design of a distillation column can be divided into the following steps:

1. Specify the degree of separation required: set product specifications.
2. Select the operating conditions: batch or continuous; operating pressure.
3. Select the type of contacting device: plates or packing.
4. Determine the stage and reflux requirements: the number of equilibrium stages.
5. Size the column: diameter, number of real stages.
6. Design the column internals: plates, distributors, packing supports.
7. Mechanical design: vessel and internal fittings.

The principal step will be to determine the stage and reflux requirements. This is a relatively simple procedure when the feed is a binary mixture, but a complex

and difficult task when the feed contains more than two components (multicomponent systems).

11.2. CONTINUOUS DISTILLATION: PROCESS DESCRIPTION

The separation of liquid mixtures by distillation depends on differences in volatility between the components. The greater the relative volatilities, the easier the separation. The basic equipment required for continuous distillation is shown in Figure 11.1. Vapour flows up the column and liquid counter-currently down the column. The vapour and liquid are brought into contact on plates, or packing. Part of the condensate from the condenser is returned to the top of the column to provide liquid flow above the feed point (reflux), and part of the liquid from the base of the column is vaporised in the reboiler and returned to provide the vapour flow.

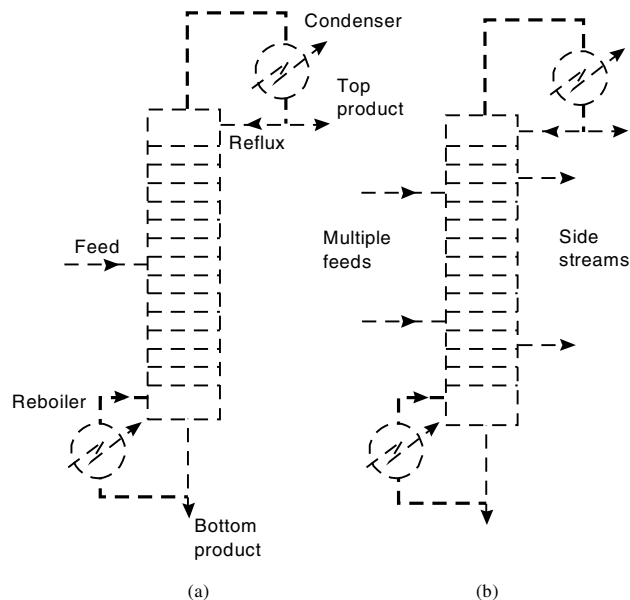


Figure 11.1. Distillation column (a) Basic column (b) Multiple feeds and side streams

In the section below the feed, the more volatile components are stripped from the liquid and this is known as the *stripping section*. Above the feed, the concentration of the more volatile components is increased and this is called the enrichment, or more commonly, the *rectifying section*. Figure 11.1a shows a column producing two product streams, referred to as *tops* and *bottoms*, from a single feed. Columns are occasionally used with more than one feed, and with side streams withdrawn at points up the column, Figure 11.1b. This does not alter the basic operation, but complicates the analysis of the process, to some extent.

If the process requirement is to strip a volatile component from a relatively non-volatile solvent, the rectifying section may be omitted, and the column would then be called a *stripping column*.

In some operations, where the top product is required as a vapour, only sufficient liquid is condensed to provide the reflux flow to the column, and the condenser is referred to as a partial condenser. When the liquid is totally condensed, the liquid returned to the column will have the same composition as the top product. In a partial condenser the reflux will be in equilibrium with the vapour leaving the condenser. Virtually pure top and bottom products can be obtained in a single column from a binary feed, but where the feed contains more than two components, only a single “pure” product can be produced, either from the top or bottom of the column. Several columns will be needed to separate a multicomponent feed into its constituent parts.

11.2.1. Reflux considerations

The reflux ratio, R , is normally defined as:

$$R = \frac{\text{flow returned as reflux}}{\text{flow of top product taken off}}$$

The number of stages required for a given separation will be dependent on the reflux ratio used.

In an operating column the effective reflux ratio will be increased by vapour condensed within the column due to heat leakage through the walls. With a well-lagged column the heat loss will be small and no allowance is normally made for this increased flow in design calculations. If a column is poorly insulated, changes in the internal reflux due to sudden changes in the external conditions, such as a sudden rain storm, can have a noticeable effect on the column operation and control.

Total reflux

Total reflux is the condition when all the condensate is returned to the column as reflux: no product is taken off and there is no feed.

At total reflux the number of stages required for a given separation is the minimum at which it is theoretically possible to achieve the separation. Though not a practical operating condition, it is a useful guide to the likely number of stages that will be needed.

Columns are often started up with no product take-off and operated at total reflux till steady conditions are attained. The testing of columns is also conveniently carried out at total reflux.

Minimum reflux

As the reflux ratio is reduced a *pinch point* will occur at which the separation can only be achieved with an infinite number of stages. This sets the minimum possible reflux ratio for the specified separation.

Optimum reflux ratio

Practical reflux ratios will lie somewhere between the minimum for the specified separation and total reflux. The designer must select a value at which the specified separation is achieved at minimum cost. Increasing the reflux reduces the number of stages required, and hence the capital cost, but increases the service requirements (steam and water) and the operating costs. The optimum reflux ratio will be that which gives the lowest annual operating cost. No hard and fast rules can be given for the selection of the design reflux ratio, but for many systems the optimum will lie between 1.2 to 1.5 times the minimum reflux ratio.

For new designs, where the ratio cannot be decided on from past experience, the effect of reflux ratio on the number of stages can be investigated using the short-cut design methods given in this chapter. This will usually indicate the best of value to use in more rigorous design methods.

At low reflux ratios the calculated number of stages will be very dependent on the accuracy of the vapour-liquid equilibrium data available. If the data are suspect a higher than normal ratio should be selected to give more confidence in the design.

11.2.2. Feed-point location

The precise location of the feed point will affect the number of stages required for a specified separation and the subsequent operation of the column. As a general rule, the feed should enter the column at the point that gives the best match between the feed composition (vapour and liquid if two phases) and the vapour and liquid streams in the column. In practice, it is wise to provide two or three feed-point nozzles located round the predicted feed point to allow for uncertainties in the design calculations and data, and possible changes in the feed composition after start-up.

11.2.3. Selection of column pressure

Except when distilling heat-sensitive materials, the main consideration when selecting the column operating-pressure will be to ensure that the dew point of the distillate is above that which can be easily obtained with the plant cooling water. The maximum, summer, temperature of cooling water is usually taken as 30°C. If this means that high pressures will be needed, the provision of refrigerated brine cooling should be considered. Vacuum operation is used to reduce the column temperatures for the distillation of heat-sensitive materials and where very high temperatures would otherwise be needed to distil relatively non-volatile materials.

When calculating the stage and reflux requirements it is usual to take the operating pressure as constant throughout the column. In vacuum columns, the column pressure drop will be a significant fraction of the total pressure and the change in pressure up the column should be allowed for when calculating the stage temperatures. This may require a trial and error calculation, as clearly the pressure drop cannot be estimated before an estimate of the number of stages is made.

11.3. CONTINUOUS DISTILLATION: BASIC PRINCIPLES

11.3.1. Stage equations

Material and energy balance equations can be written for any stage in a multistage process.

Figure 11.2 shows the material flows into and out of a typical stage n in a distillation column. The equations for this stage are set out below, for any component i .

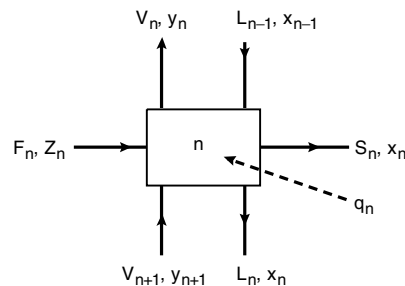


Figure 11.2. Stage flows

material balance

$$V_{n+1}y_{n+1} + L_{n-1}x_{n-1} + F_n z_n = V_n y_n + L_n x_n + S_n x_n \quad (11.1)$$

energy balance

$$V_{n+1}H_{n+1} + L_{n-1}h_{n-1} + Fh_f + q_n = V_n H_n + L_n h_n + S_n h_n \quad (11.2)$$

where V_n = vapour flow from the stage,

V_{n+1} = vapour flow into the stage from the stage below,

L_n = liquid flow from the stage,

L_{n-1} = liquid flow into the stage from the stage above,

F_n = any feed flow into the stage,

S_n = any side stream from the stage,

q_n = heat flow into, or removal from, the stage,

n = any stage, numbered from the top of the column,

z = mol fraction of component i in the feed stream (note, feed may be two-phase),

x = mol fraction of component i in the liquid streams,

y = mol fraction component i in the vapour streams,

H = specific enthalpy vapour phase,

h = specific enthalpy liquid phase,

h_f = specific enthalpy feed (vapour + liquid).

All flows are the total stream flows (mols/unit time) and the specific enthalpies are also for the total stream (J/mol).

It is convenient to carry out the analysis in terms of “equilibrium stages”. In an equilibrium stage (theoretical plate) the liquid and vapour streams leaving the stage are taken to be in equilibrium, and their compositions are determined by the vapour-liquid equilibrium relationship for the system (see Chapter 8). In terms of equilibrium constants:

$$y_i = K_i x_i \quad (11.3)$$

The performance of real stages is related to an equilibrium stage by the concept of plate efficiencies for plate contactors, and “height of an equivalent theoretical plate” for packed columns.

In addition to the equations arising from the material and energy balances over a stage, and the equilibrium relationships, there will be a fourth relationship, the summation equation for the liquid and vapour compositions:

$$\sum x_{i,n} = \sum y_{i,n} = 1.0 \quad (11.4)$$

These four equations are the so-called MESH equations for the stage: Material balance, Equilibrium, Summation and Heat (energy) balance, equations. MESH equations can be written for each stage, and for the reboiler and condenser. The solution of this set of equations forms the basis of the rigorous methods that have been developed for the analysis for staged separation processes.

11.3.2. Dew points and bubble points

To estimate the stage, and the condenser and reboiler temperatures, procedures are required for calculating dew and bubble points. By definition, a saturated liquid is at its bubble point (any rise in temperature will cause a bubble of vapour to form), and a saturated vapour is at its dew point (any drop in temperature will cause a drop of liquid to form).

Dew points and bubble points can be calculated from a knowledge of the vapour-liquid equilibrium for the system. In terms of equilibrium constants, the bubble point is defined by the equation:

$$\text{bubble point: } \sum y_i = \sum K_i x_i = 1.0 \quad (11.5a)$$

$$\text{and dew point: } \sum x_i = \sum \frac{y_i}{K_i} = 1.0 \quad (11.5b)$$

For multicomponent mixtures the temperature that satisfies these equations, at a given system pressure, must be found by trial and error.

For binary systems the equations can be solved more readily because the component compositions are not independent; fixing one fixes the other.

$$y_a = 1 - y_b \quad (11.6a)$$

$$x_a = 1 - x_b \quad (11.6b)$$

Bubble- and dew-point calculations are illustrated in Example 11.9.

11.3.3. Equilibrium flash calculations

In an equilibrium flash process a feed stream is separated into liquid and vapour streams at equilibrium. The composition of the streams will depend on the quantity of the feed vaporised (flashed). The equations used for equilibrium flash calculations are developed below and a typical calculation is shown in Example 11.1.

Flash calculations are often needed to determine the condition of the feed to a distillation column and, occasionally, to determine the flow of vapour from the reboiler, or condenser if a partial condenser is used.

Single-stage flash distillation processes are used to make a coarse separation of the light components in a feed; often as a preliminary step before a multicomponent distillation column, as in the distillation of crude oil.

Figure 11.3 shows a typical equilibrium flash process. The equations describing this process are:

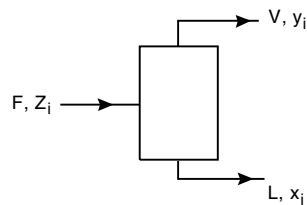


Figure 11.3. Flash distillation

Material balance, for any component, i

$$Fz_i = Vy_i + Lx_i \quad (11.7)$$

Energy balance, total stream enthalpies:

$$Fh_f = VH + Lh \quad (11.8)$$

If the vapour-liquid equilibrium relationship is expressed in terms of equilibrium constants, equation 11.7 can be written in a more useful form:

$$\begin{aligned} Fz_i &= VK_ix_i + Lx_i \\ &= Lx_i \left[\frac{V}{L}K_i + 1 \right] \end{aligned}$$

from which

$$L = \sum_i \frac{Fz_i}{\left[\frac{VK_i}{L} + 1 \right]} \quad (11.9)$$

and, similarly,

$$V = \sum_i \frac{Fz_i}{\left[\frac{L}{VK_i} + 1 \right]} \quad (11.10)$$

The groups incorporating the liquid and vapour flow-rates and the equilibrium constants have a general significance in separation process calculations.

The group L/VK_i is known as the absorption factor A_i , and is the ratio of the mols of any component in the liquid stream to the mols in the vapour stream.

The group VK_i/L is called the stripping factor S_i , and is the reciprocal of the absorption factor.

Efficient techniques for the solution of the trial and error calculations necessary in multicomponent flash calculations are given by several authors; Hengstebeck (1976) and King (1980).

Example 11.1

A feed to a column has the composition given in the table below, and is at a pressure of 14 bar and a temperature of 60°C. Calculate the flow and composition of the liquid and vapour phases. Take the equilibrium data from the Depriester charts given in Chapter 8.

		kmol/h	z_i
Feed	ethane (C ₂)	20	0.25
	propane (C ₃)	20	0.25
	isobutane (iC ₄)	20	0.25
	n-pentane (nC ₅)	20	0.25

Solution

For two phases to exist the flash temperature must lie between the bubble point and dew point of the mixture.

From equations 11.5a and 11.5b:

$$\sum K_i z_i > 1.0$$

$$\sum \frac{z_i}{K_i} > 1.0$$

Check feed condition

	K_i	$K_i z_i$	z_i/K_i
C ₂	3.8	0.95	0.07
C ₃	1.3	0.33	0.19
iC ₄	0.43	0.11	0.58
nC ₅	0.16	0.04	1.56
		Σ 1.43	Σ 2.40

therefore the feed is a two phase mixture.

Flash calculation

	K_i	Try $L/V = 1.5$		Try $L/V = 3.0$	
		$A_i = L/VK_i$	$V_i = Fz_i/(1 + A_i)$	A_i	V_i
C ₂	3.8	0.395	14.34	0.789	11.17
C ₃	1.3	1.154	9.29	2.308	6.04
iC ₄	0.43	3.488	4.46	6.977	2.51
nC ₅	0.16	9.375	1.93	18.750	1.01
		$V_{\text{calc}} = 30.02$		$V_{\text{calc}} = 20.73$	
		$L/V = \frac{80 - 30.02}{30.02} = 1.67$		$L/V = 2.80$	

Hengstebeck's method is used to find the third trial value for L/V . The calculated values are plotted against the assumed values and the intercept with a line at 45° (calculated = assumed) gives the new trial value, 2.4.

	Try $L/V = 2.4$			
	A_i	V_i	$y_i = V_i/V$	$x_i = (Fz_i - V_i)/L$
C ₂	0.632	12.26	0.52	0.14
C ₃	1.846	7.03	0.30	0.23
iC ₄	5.581	3.04	0.13	0.30
nC ₅	15.00	1.25	0.05	0.33
		$V_{\text{cal}} = 23.58$	1.00	1.00
$L = 80 - 23.58 = 56.42$ kmol/h,				
L/V calculated = $56.42/23.58 = 2.39$ close enough to the assumed value of 2.4.				

Adiabatic flash

In many flash processes the feed stream is at a higher pressure than the flash pressure and the heat for vaporisation is provided by the enthalpy of the feed. In this situation the flash temperature will not be known and must be found by trial and error. A temperature must be found at which both the material and energy balances are satisfied.

11.4. DESIGN VARIABLES IN DISTILLATION

It was shown in Chapter 1 that to carry out a design calculation the designer must specify values for a certain number of independent variables to define the problem completely, and that the ease of calculation will often depend on the judicious choice of these design variables.

In manual calculations the designer can use intuition in selecting the design variables and, as he proceeds with the calculation, can define other variables if it becomes clear that

the problem is not sufficiently defined. He can also start again with a new set of design variables if the calculations become tortuous. When specifying a problem for a computer method it is essential that the problem is completely and sufficiently defined.

In Chapter 1 it was shown that the number of independent variables for any problem is equal to the difference between the total number of variables and the number of linking equations and other relationships. Examples of the application of this formal procedure for determining the number of independent variables in separation process calculations are given by Gilliland and Reed (1942) and Kwauk (1956). For a multistage, multicomponent, column, there will be a set of material and enthalpy balance equations and equilibrium relationships for each stage (the MESH equations), and for the reboiler and condenser; for each component. If there are more than a few stages the task of counting the variables and equations becomes burdensome and mistakes are very likely to be made. A simpler, more practical, way to determine the number of independent variables is the "description rule" procedure given by Hanson *et al.* (1962). Their description rule states that to determine a separation process completely the number of independent variables which must be set (by the designer) will equal the number that are set in the construction of the column or that can be controlled by external means in its operation. The application of this rule requires the designer to visualise the column in operation and list the number of variables fixed by the column construction; those fixed by the process; and those that have to be controlled for the column to operate steadily and produce product within specification. The method is best illustrated by considering the operation of the simplest type of column: with one feed, no side streams, a total condenser, and a reboiler. The construction will fix the number of stages above and below the feed point (two variables). The feed composition and total enthalpy will be fixed by the processes upstream ($1 + (n - 1)$ variables, where n is the number of components). The feed rate, column pressure and condenser and reboiler duties (cooling water and steam flows) will be controlled (four variables).

$$\text{Total number of variables fixed} = 2 + 1 + (n - 1) + 4 = \underline{\underline{n + 6}}$$

To design the column this number of variables must be specified completely to define the problem, but the same variables need not be selected.

Typically, in a design situation, the problem will be to determine the number of stages required at a specified reflux ratio and column pressure, for a given feed, and with the product compositions specified in terms of two key components and one product flow-rate. Counting up the number of variables specified it will be seen that the problem is completely defined:

Feed flow, composition, enthalpy	= $2 + (n - 1)$
Reflux (sets q_c)	= 1
Key component compositions, top and bottom	= 2
Product flow	= 1
Column pressure	= 1
	<hr style="width: 100%; border: 0.5px solid black;"/>
	<u><u>$n + 6$</u></u>

Note: specifying $(n - 1)$ component compositions completely defines the feed composition as the fractions add up to 1.

In theory any $(n + 6)$ independent variables could have been specified to define the problem, but it is clear that the use of the above variables will lead to a straightforward solution of the problem.

When replacing variables identified by the application of the description rule it is important to ensure that those selected are truly independent, and that the values assigned to them lie within the range of possible, practical, values.

The number of independent variables that have to be specified to define a problem will depend on the type of separation process being considered. Some examples of the application of the description rule to more complex columns are given by Hanson *et al.* (1962).

11.5. DESIGN METHODS FOR BINARY SYSTEMS

A good understanding of the basic equations developed for binary systems is essential to the understanding of distillation processes.

The distillation of binary mixtures is covered thoroughly in Volume 2, Chapter 11, and the discussion in this section is limited to a brief review of the most useful design methods. Though binary systems are usually considered separately, the design methods developed for multicomponent systems (Section 11.6) can obviously also be used for binary systems. With binary mixtures fixing the composition of one component fixes the composition of the other, and iterative procedures are not usually needed to determine the stage and reflux requirements; simple graphical methods are normally used.

11.5.1. Basic equations

Sorel (1899) first derived and applied the basic stage equations to the analysis of binary systems. Figure 11.4*a* shows the flows and compositions in the top part of a column. Taking the system boundary to include the stage n and the condenser, gives the following equations:

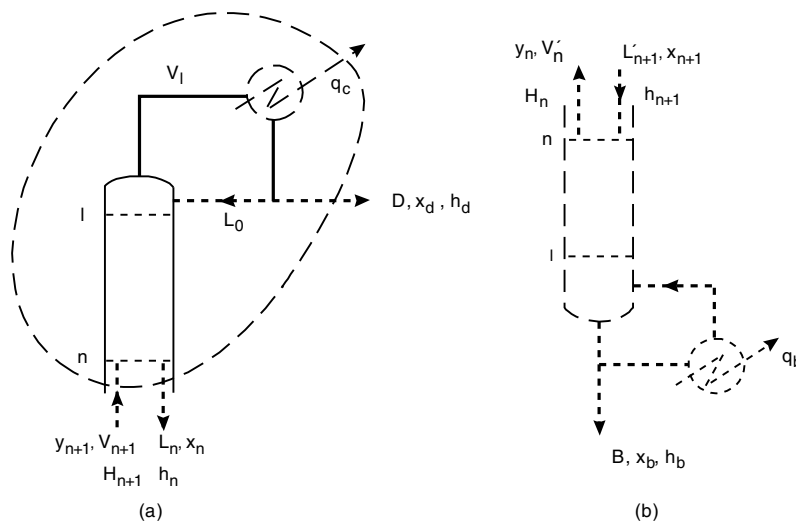


Figure 11.4. Column flows and compositions (a) Above feed (b) Below feed

Material balance

$$\text{total flows} \quad V_{n+1} = L_n + D \quad (11.11)$$

$$\text{for either component} \quad V_{n+1} y_{n+1} = L_n x_n + D x_d \quad (11.12)$$

Energy balance

$$\text{total stream enthalpies} \quad V_{n+1} H_{n+1} = L_n h_n + D h_d + q_c \quad (11.13)$$

where q_c is the heat removed in the condenser.

Combining equations 11.11 and 11.12 gives

$$y_{n+1} = \frac{L_n}{L_n + D} x_n + \frac{D}{L_n + D} x_d \quad (11.14)$$

Combining equations 11.11 and 11.13 gives

$$V_{n+1} H_{n+1} = (L_n + D) H_{n+1} = L_n h_n + D h_d + q_c \quad (11.15)$$

Analogous equations can be written for the stripping section, Figure 11.6*b*.

$$x_{n+1} = \frac{V'_n}{V'_n + B} y_n + \frac{B}{V'_n + B} x_b \quad (11.16)$$

and

$$L'_{n+1} h_{n+1} = (V'_n + B) h_{n+1} = V'_n H_n + B h_b - q_b \quad (11.17)$$

At constant pressure, the stage temperatures will be functions of the vapour and liquid compositions only (dew and bubble points) and the specific enthalpies will therefore also be functions of composition

$$H = f(y) \quad (11.18a)$$

$$h = f(x) \quad (11.18b)$$

Lewis-Sorel method (equimolar overflow)

For most distillation problems a simplifying assumption, first proposed by Lewis (1909), can be made that eliminates the need to solve the stage energy-balance equations. The molar liquid and vapour flow rates are taken as constant in the stripping and rectifying sections. This condition is referred to as equimolar overflow: the molar vapour and liquid flows from each stage are constant. This will only be true where the component molar latent heats of vaporisation are the same and, together with the specific heats, are constant over the range of temperature in the column; there is no significant heat of mixing; and the heat losses are negligible. These conditions are substantially true for practical systems when the components form near-ideal liquid mixtures.

Even when the latent heats are substantially different the error introduced by assuming equimolar overflow to calculate the number of stages is usually small, and acceptable.

With equimolar overflow equations 11.14 and 11.16 can be written without the subscripts to denote the stage number:

$$y_{n+1} = \frac{L}{L+D}x_n + \frac{D}{L+D}x_d \quad (11.19)$$

$$x_{n+1} = \frac{V'}{V'+B}y_n + \frac{B}{V'+B}x_b \quad (11.20)$$

where L = the constant liquid flow in the rectifying section = the reflux flow, L_0 , and V' is the constant vapour flow in the stripping section.

Equations 11.19 and 11.20 can be written in an alternative form:

$$y_{n+1} = \frac{L}{V}x_n + \frac{D}{V}x_d \quad (11.21)$$

$$y_n = \frac{L'}{V'}x_{n+1} - \frac{B}{V'}x_b \quad (11.22)$$

where V is the constant vapour flow in the rectifying section = $(L + D)$; and L' is the constant liquid flow in the stripping section = $V' + B$.

These equations are linear, with slopes L/V and L'/V' . They are referred to as *operating lines*, and give the relationship between the liquid and vapour compositions between stages. For an equilibrium stage, the compositions of the liquid and vapour streams leaving the stage are given by the equilibrium relationship.

11.5.2. McCabe-Thiele method

Equations 11.21 and 11.22 and the equilibrium relationship are conveniently solved by the graphical method developed by McCabe and Thiele (1925). The method is discussed fully in Volume 2. A simple procedure for the construction of the diagram is given below and illustrated in Example 11.2.

Procedure

Refer to Figure 11.5, all compositions are those of the more volatile component.

1. Plot the vapour-liquid equilibrium curve from data available at the column operating pressure. In terms of relative volatility:

$$y = \frac{\alpha x}{1 + (\alpha - 1)x} \quad (11.23)$$

where α is the geometric average relative volatility of the lighter (more volatile) component with respect to the heavier component (less volatile).

It is usually more convenient, and less confusing, to use equal scales for the x and y axes.

2. Make a material balance over the column to determine the top and bottom compositions, x_d and x_b , from the data given.
3. The top and bottom operating lines intersect the diagonal at x_d and x_b respectively; mark these points on the diagram.
4. The point of intersection of the two operating lines is dependent on the phase condition of the feed. The line on which the intersection occurs is called the *q line* (see Volume 2). The *q line* is found as follows:

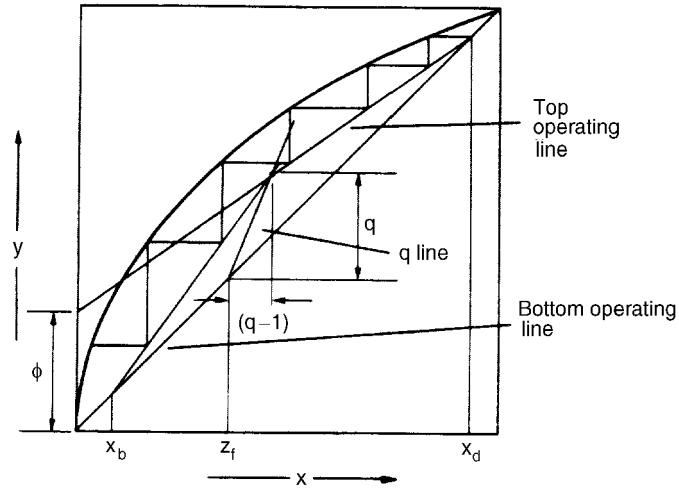


Figure 11.5. McCabe-Thiele diagram

- (i) calculate the value of the ratio q given by

$$q = \frac{\text{heat to vaporise 1 mol of feed}}{\text{molar latent heat of feed}}$$

- (ii) plot the q line, slope = $q/(q-1)$, intersecting the diagonal at z_f (the feed composition).

5. Select the reflux ratio and determine the point where the top operating line extended cuts the y axis:

$$\phi = \frac{x_d}{1 + R} \quad (11.24)$$

6. Draw in the top operating line, from x_d on the diagonal to ϕ .
7. Draw in the bottom operating line; from x_b on the diagonal to the point of intersection of the top operating line and the q line.
8. Starting at x_d or x_b , step off the number of stages.

Note: The feed point should be located on the stage closest to the intersection of the operating lines.

The reboiler, and a partial condenser if used, act as equilibrium stages. However, when designing a column there is little point in reducing the estimated number of stages to account for this; they can be considered additional factors of safety.

The efficiency of real contacting stages can be accounted for by reducing the height of the steps on the McCabe-Thiele diagram, see diagram Figure 11.6. Stage efficiencies are discussed in Section 11.10.

The McCabe-Thiele method can be used for the design of columns with side streams and multiple feeds. The liquid and vapour flows in the sections between the feed and take-off points are calculated and operating lines drawn for each section.

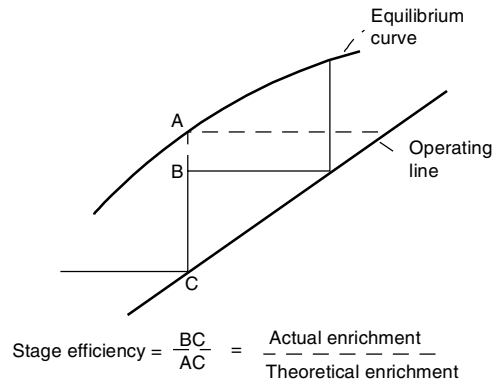


Figure 11.6. Stage efficiency

Stage vapour and liquid flows not constant

The McCabe-Thiele method can be used when the condition of equimolar overflow cannot be assumed, but the operating lines will not then be straight. They can be drawn by making energy balances at a sufficient number of points to determine the approximate slope of the lines; see Hengstebeck (1976). Alternatively the more rigorous graphical method of Ponchon and Savarit derived in Volume 2 can be used. Nowadays, it should rarely be necessary to resort to complex graphical methods when the simple McCabe-Thiele diagram is not sufficiently accurate, as computer programs will normally be available for the rigorous solution of such problems.

11.5.3. Low product concentrations

When concentrations of the more volatile component of either product is very low the steps on the McCabe-Thiele diagram become very small and difficult to plot. This problem can be overcome by replotting the top or bottom sections to a larger scale, or on log-log paper. In a log plot the operating line will not be straight and must be drawn by plotting points calculated using equations 11.21 and 11.22. This technique is described by Alleva (1962) and is illustrated in Example 11.2.

If the operating and equilibrium lines are straight, and they usually can be taken as such when the concentrations are small, the number of stages required can be calculated using the equations given by Robinson and Gilliland (1950).

For the stripping section:

$$N_s^* = \frac{\log \left[\frac{\left(\frac{K'}{s'} - 1 \right) \left(\frac{x_r'}{x_b} - 1 \right)}{\frac{1}{s'}(K' - 1)} + 1 \right]}{\log \left(\frac{K'}{s'} \right)} + 1 \quad (11.25)$$

where N_s^* = number of ideal stages required from x_b to some reference point x_r' ,
 x_b = mol fraction of the more volatile component in the bottom product,

- x'_r = mol fraction of more volatile component at the reference point,
 s' = slope of the bottom operating line,
 K' = equilibrium constant for the more volatile component.

For the rectifying section:

$$N_r^* = \frac{\log \left[\frac{(1-s) + x_r/x_d(s-K)}{1-K} \right]}{\log \left(\frac{s}{K} \right)} - 1 \quad (11.26)$$

- where N_r^* = number of stages required from some reference point x_r to the x_d ,
 x_d = mol fraction of the *least volatile* component in the top product,
 x_r = mol fraction of *least volatile* component at reference point,
 K = equilibrium constant for the *least volatile* component,
 s = slope of top operating line.

Note: at low concentrations $K = \alpha$.

The use of these equations is illustrated in Example 11.3.

Example 11.2

Acetone is to be recovered from an aqueous waste stream by continuous distillation. The feed will contain 10 per cent w/w acetone. Acetone of at least 98 per cent purity is wanted, and the aqueous effluent must not contain more than 50 ppm acetone. The feed will be at 20°C. Estimate the number of ideal stages required.

Solution

There is no point in operating this column at other than atmospheric pressure. The equilibrium data available for the acetone-water system were discussed in Chapter 8, Section 8.4.

The data of Kojima *et al.* will be used.

Mol fraction x , liquid	0.00	0.05	0.10	0.15	0.20	0.25	0.30
Acetone y , vapour	0.00	0.6381	0.7301	0.7716	0.7916	0.8034	0.8124
bubble point °C	100.0	74.80	68.53	65.26	63.59	62.60	61.87

x	0.35	0.40	0.45	0.50	0.55	0.60	0.65
y	0.8201	0.8269	0.8376	0.8387	0.8455	0.8532	0.8615
°C	61.26	60.75	60.35	59.95	59.54	59.12	58.71

x	0.70	0.75	0.80	0.85	0.90	0.95
y	0.8712	0.8817	0.8950	0.9118	0.9335	0.9627
°C	58.29	57.90	57.49	57.08	56.68	56.30

The equilibrium curve can be drawn with sufficient accuracy to determine the stages above the feed by plotting the concentrations at increments of 0.1. The diagram would normally be plotted at about twice the size of Figure 11.7.

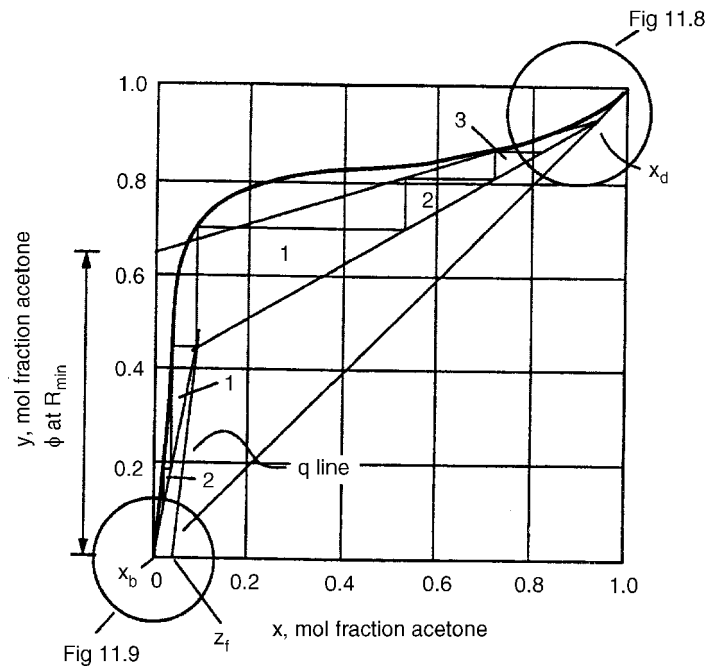


Figure 11.7. McCabe-Thiele plot, Example 11.2

Molecular weights, acetone 58, water 18

$$\text{Mol fractions acetone feed} = \frac{\frac{10}{58}}{\frac{10}{58} + \frac{90}{18}} = 0.033$$

$$\text{top product} = \frac{\frac{98}{58}}{\frac{98}{58} + \frac{2}{18}} = 0.94$$

$$\text{bottom product} = 50 \times 10^{-6} \times \frac{18}{58} = 15.5 \times 10^{-6}$$

Feed condition (q-line)

Bubble point of feed (interpolated) = 83°C

Latent heats, water 41,360, acetone 28,410 J/mol

Mean specific heats, water 75.3, acetone 128 J/mol °C

Latent heat of feed = $28,410 \times 0.033 + (1 - 0.033) 41,360 = 40,933$ J/mol

Specific heat of feed = $(0.033 \times 128) + (1 - 0.033) 75.3 = 77.0$ J/mol °C

Heat to vaporise 1 mol of feed = $(83 - 20) 77.0 + 40,933 = 45,784$ J

$$q = \frac{45,784}{40,933} = 1.12$$

$$\text{Slope of } q \text{ line} = \frac{1.12}{1.12 - 1} = 9.32$$

For this problem the condition of minimum reflux occurs where the top operating line just touches the equilibrium curve at the point where the q line cuts the curve.

From the Figure 11.7,

$$\phi \text{ for the operating line at minimum reflux} = 0.65$$

From equation 11.24, $R_{\min} = 0.94/0.65 - 1 = 0.45$

Take $R = R_{\min} \times 3$

As the flows above the feed point will be small, a high reflux ratio is justified; the condenser duty will be small.

$$\text{At } R = 3 \times 0.45 = 1.35, \quad \phi = \frac{0.94}{1 + 1.35} = 0.4$$

For this problem it is convenient to step the stages off starting at the intersection of the operating lines. This gives three stages above the feed up to $y = 0.8$. The top section is drawn to a larger scale, Figure 11.8, to determine the stages above $y = 0.8$: three to four stages required; total stages above the feed 7.

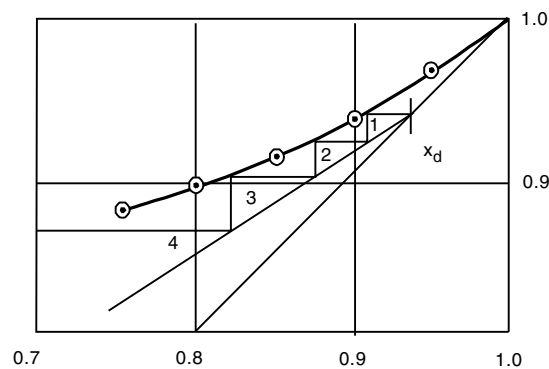


Figure 11.8. Top section enlarged

Below the feed, one stage is required down to $x = 0.04$. A log-log plot is used to determine the stages below this concentration. Data for log-log plot:

operating line slope, from Figure 11.7 = $0.45/0.09 = 5.0$

operating line equation, $y = 4.63(x - x_b) + x_b$

$$= 5.0x - 62.0 \times 10^{-6}$$

equilibrium line slope, from v-1-e data $= 0.6381/0.05 = 12.8$

	$x = 4 \times 10^{-2}$	10^{-3}	10^{-4}	4×10^{-5}	2×10^{-5}
Equilibrium line $y =$	0.51	1.3×10^{-2}	1.3×10^{-3}	5.1×10^{-4}	2.6×10^{-4}
Operating line $y =$	0.20	4.9×10^{-3}	4.4×10^{-4}	1.4×10^{-4}	3.8×10^{-5}

From Figure 11.9, number of stages required for this section = 8

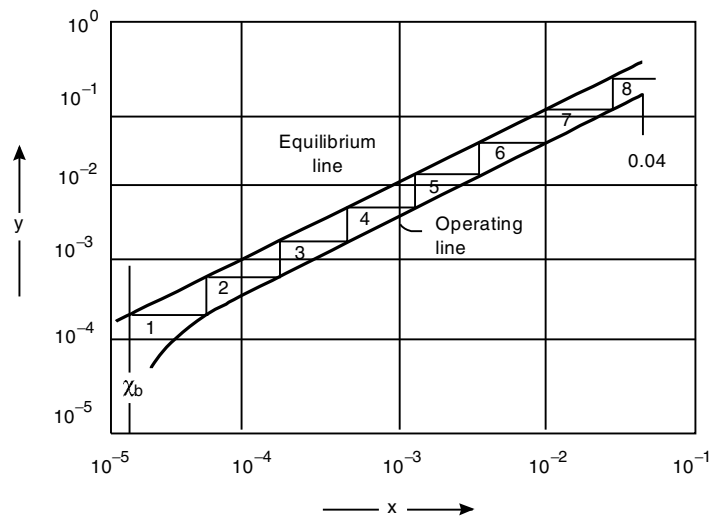


Figure 11.9. Log-log plot of McCabe-Thiele diagram

Total number of stages below feed = 9

Total stages = $7 + 9 = \underline{\underline{16}}$

Example 11.3

For the problem specified in Example 11.2, estimate the number of ideal stages required below an acetone concentration of 0.04 (more volatile component), using the Robinson-Gilliland equation.

Solution

From the McCabe-Thiele diagram in Example 11.2:

slope of bottom operating line, $s' = 5.0$

slope of equilibrium line, $K' = 12.8$

$$x_b = 15.5 \times 10^{-6}$$

$$N_s^* = \frac{\log \left[\frac{\left(\frac{12.8}{5.0} - 1 \right) \left(\frac{0.04}{15.5 \times 10^{-6}} - 1 \right)}{\left(\frac{1}{5.0} \right) (12.8 - 1)} + 1 \right]}{\log \left(\frac{12.8}{5.0} \right)} + 1 = 8.9, \underline{\underline{\text{say } 9}} \quad (11.25)$$

11.5.4. The Smoker equations

Smoker (1938) derived an analytical equation that can be used to determine the number of stages when the relative volatility is constant. Though his method can be used for any problem for which the relative volatilities in the rectifying and stripping sections can be taken as constant, it is particularly useful for problems where the relative volatility is low; for example, in the separation of close boiling isomers. If the relative volatility is close to one, the number of stages required will be very large, and it will be impractical to draw a McCabe-Thiele diagram. The derivation of the equations are outlined below and illustrated in Example 11.4.

Derivation of the equations:

A straight operating line can be represented by the equation:

$$y = sx + c \quad (11.27)$$

and in terms of relative volatility the equilibrium values of y are given by:

$$y = \frac{\alpha x}{1 + (\alpha - 1)x} \quad (\text{equation 11.23})$$

Eliminating y from these equations gives a quadratic in x :

$$s(\alpha - 1)x^2 + [s + b(\alpha - 1) - \alpha]x + b = 0 \quad (11.28)$$

For any particular distillation problem equation 11.28 will have only one real root k between 0 and 1

$$s(\alpha - 1)k^2 + [s + b(\alpha - 1) - \alpha]k + b = 0 \quad (11.29)$$

k is the value of the x ordinate at the point where the extended operating lines intersect the vapour-liquid equilibrium curve. Smoker shows that the number of stages required is given by the equation:

$$N = \log \left[\frac{x_0^*(1 - \beta x_n^*)}{x_n^*(1 - \beta x_0^*)} \right] / \log \left(\frac{\alpha}{sc^2} \right) \quad (11.30)$$

where

$$\beta = \frac{sc(\alpha - 1)}{\alpha - sc^2} \quad (11.31)$$

N = number of stages required to effect the separation represented by the concentration change from

$$x_n^* \text{ to } x_0^*; x^* = (x - k) \text{ and } x_0^* > x_n^* \quad (11.32)$$

$$c = 1 + (\alpha - 1)k$$

s = slope of the operating line between x_n^* and x_0^* ,
 α = relative volatility, assumed constant over x_n^* to x_0^* .

For a column with a single feed and no side streams:

Rectifying section

$$x_0^* = x_d - k \quad (11.33)$$

$$x_n^* = z_f - k \quad (11.34)$$

$$s = \frac{R}{R+1} \quad (11.35)$$

$$b = \frac{x_d}{R+1} \quad (11.36)$$

Stripping section

$$x_0^* = z_f - k \quad (11.37)$$

$$x_n^* = x_b - k \quad (11.38)$$

$$s = \frac{Rz_f + x_d - (R+1)x_b}{(R+1)(z_f - x_b)} \quad (11.39)$$

$$b = \frac{(z_f - x_d)x_b}{(R+1)(z_f - x_b)} \quad (11.40)$$

If the feed stream is not at its bubble point, z_f is replaced by the value of x at the intersection of operating lines, given by

$$z_f^* = \frac{b + \frac{z_f}{q-1}}{\frac{q}{q-1} - s} \quad (11.41)$$

All compositions for the more volatile component.

Example 11.4

A column is to be designed to separate a mixture of ethylbenzene and styrene. The feed will contain 0.5 mol fraction styrene, and a styrene purity of 99.5 per cent is required, with a recovery of 85 per cent. Estimate the number of equilibrium stages required at a reflux ratio of 8. Maximum column bottom pressure 0.20 bar.

Solution

Ethylbenzene is the more volatile component.

$$\text{Antoine equations, ethylbenzene, } \ln P^\circ = 9.386 - \frac{3279.47}{T - 59.95}$$

$$\text{styrene } \ln P^\circ = 9.386 - \frac{3328.57}{T - 63.72}$$

P bar, T Kelvin

Material balance, basis 100 kmol feed:

at 85 per cent recovery, styrene in bottoms = $50 \times 0.85 = 42.5$ kmol

at 99.5 per cent purity, ethylbenzene in bottoms = $\frac{42.5}{99.5} \times 0.5 = 0.21$ kmol

ethylbenzene in the tops = $50 - 0.21 = 49.79$ kmol

styrene in tops = $50 - 42.5 = 7.5$ kmol

mol fraction ethylbenzene in tops = $\frac{49.79}{49.79 + 7.5} = 0.87$

$z_f = 0.5$, $x_b = 0.005$, $x_d = 0.87$

Column bottom temperature, from Antoine equation for styrene

$$\ln 0.2 = 9.386 - \frac{3328.57}{T - 63.72}$$

$$T = 366 \text{ K}, 93.3^\circ\text{C}$$

At 93.3°C , vapour pressure of ethylbenzene

$$\ln P^\circ = 9.386 - \frac{3279.47}{366.4 - 59.95} = 0.27 \text{ bar}$$

$$\text{Relative volatility} = \frac{P^\circ \text{ ethylbenzene}}{P^\circ \text{ styrene}} = \frac{0.27}{0.20} = 1.35$$

The relative volatility will change as the compositions and (particularly for a vacuum column) the pressure changes up the column. The column pressures cannot be estimated until the number of stages is known; so as a first trial the relative volatility will be taken as constant, at the value determined by the bottom pressure.

Rectifying section

$$s = \frac{8}{8 + 1} = 0.89 \quad (11.35)$$

$$b = \frac{0.87}{8 + 1} = 0.097 \quad (11.36)$$

$$0.89(1.35 - 1)k^2 + [0.89 + 0.097(1.35 - 1) - 1.35]k + 0.097 = 0 \quad (11.29)$$

$$k = 0.290$$

$$x_0^* = 0.87 - 0.29 = 0.58 \quad (11.33)$$

$$x_n^* = 0.50 - 0.29 = 0.21 \quad (11.34)$$

$$c = 1 + (1.35 - 1)0.29 = 1.10 \quad (11.32)$$

$$\beta = \frac{0.89 \times 1.10(1.35 - 1)}{1.35 - 0.89 \times 1.1^2} = 1.255 \quad (11.31)$$

$$N = \log \left[\frac{0.58(1 - 1.255 \times 0.21)}{0.21(1 - 1.255 \times 0.58)} \right] / \log \left(\frac{1.35}{0.89 \times 1.1^2} \right) \quad (11.30)$$

$$= \frac{\log 7.473}{\log 1.254} = 8.87, \text{ say } \underline{\underline{9}}$$

Stripping section, feed taken as at its bubble point

$$s = \frac{8 \times 0.5 + 0.87 - (8 + 1)0.005}{(8 + 1)(0.5 - 0.005)} = 1.084 \quad (11.39)$$

$$b = \frac{(0.5 - 0.87)0.005}{(8 + 1)(0.5 - 0.005)} = -4.15 \times 10^{-4} \text{ (essentially zero)} \quad (11.40)$$

$$1.084(1.35 - 1)k^2 + [1.084 - 4.15 \times 10^{-4}(1.35 - 1) - 1.35]k - 4.15 \times 10^{-4} = 0$$

$$k = 0.702 \quad (11.29)$$

$$x_0^* = 0.5 - 0.702 = -0.202 \quad (11.37)$$

$$x_n^* = 0.005 - 0.702 = -0.697 \quad (11.38)$$

$$c = 1 + (1.35 - 1)0.702 = 1.246 \quad (11.32)$$

$$\beta = \frac{1.084 \times 1.246(1.35 - 1)}{1.35 - 1.084 \times 1.246^2} = -1.42 \quad (11.31)$$

$$N = \log \left[\frac{-0.202(1 - 0.697 \times 1.42)}{-0.697(1 - 0.202 \times 1.42)} \right] / \log \left(\frac{1.35}{1.084 \times 1.246^2} \right) \quad (11.30)$$

$$= \frac{\log[4.17 \times 10^{-3}]}{\log 0.8} = 24.6, \text{ say } \underline{\underline{25}}$$

11.6. MULTICOMPONENT DISTILLATION: GENERAL CONSIDERATIONS

The problem of determining the stage and reflux requirements for multicomponent distillations is much more complex than for binary mixtures. With a multicomponent mixture, fixing one component composition does not uniquely determine the other component compositions and the stage temperature. Also when the feed contains more than two components it is not possible to specify the complete composition of the top and bottom products independently. The separation between the top and bottom products is specified by setting limits on two "key" components, between which it is desired to make the separation.

The complexity of multicomponent distillation calculations can be appreciated by considering a typical problem. The normal procedure is to solve the MESH equations (Section 11.3.1) stage-by-stage, from the top and bottom of the column toward the feed point. For such a calculation to be exact, the compositions obtained from both the bottom-up and top-down calculations must mesh at the feed point and match the feed composition. But the calculated compositions will depend on the compositions assumed for the top and bottom products at the commencement of the calculations. Though it is possible to